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EFFECTS OF TEMPERATURE AND HYDROGEN ON FATIGUE PROPERTIES ON 304L

Romain Chochoy¹, Pierre Osmond², Denis Bertheau¹, Guillaume Benoit¹, Daniella Guedes Sales²,
Gouenou Girardin², Gilbert Hénaff¹

¹Institut P' UPR 3346 CNRS – ENSMA – Université de Poitiers, Téléport 2, 1 avenue Clément Ader, 86961
Futuroscope Chasseneuil cedex, France

²Cetim, 74 route de la Jonelière – CS 50814, 44308 Nantes Cedex 3, France

ABSTRACT

The design of liquid hydrogen tanks requires a thorough understanding of the factors influencing fatigue crack initiation and propagation at cryogenic temperatures in materials used in hydrogen environments. To gain a more precise understanding of the impact of hydrogen on the fatigue properties of austenitic stainless-steel alloys, this study examines its effect on fatigue strength and behaviour at different temperatures. Indeed, low temperatures raises the role of changes in the microstructure in relation with hydrogen embrittlement of these steels. The study concludes that hydrogen affects fatigue strength at ambient temperature. As for the evolution of the ratcheting deformation, no difference was noted between an air and hydrogen environment. These results will allow further steps to compare the effect of external hydrogen (in-situ tests) with that in solution (ex-situ tests).

Keywords: Hydrogen embrittlement; Austenitic stainless steels; Crack Initiation; Crack propagation; Fatigue life; Ratcheting deformation

1. INTRODUCTION

The demand for hydrogen as an energy carrier is currently high, especially in industries that emit greenhouse gases, such as aviation and the energy sector. This is due to the need for decarbonisation. In the development of a sustainable hydrogen industry, storage has proven to be a critical factor in making the use of hydrogen viable. Liquid hydrogen storage is preferred for storing large quantities of hydrogen due to its greater volumetric capacity compared to hydrogen in gaseous form. The design of the tanks is the main challenge in developing this technology. Indeed numerous studies have shown that hydrogen has a significant impact on the monotonic tensile behaviour of these steels at varying exposure temperatures [1,2]. Hydrogen embrittlement (HE) reaches a maximum around 190K (-83°C) and has a reduced impact at temperatures below 150K. These results suggest that below 190K, the diffusivity of hydrogen in the material controls HE, while above this temperature hydrogen embrittlement might be affected by the change in microstructure (martensitic transformation). However, the study of the damage mechanisms in austenitic stainless steels in low-temperature fatigue remains relatively unexplored. According to T. Ogata, the impact of hydrogen on the behaviour of steels over large numbers of cycles appears to be limited [3]. Therefore, this work aims to assess the influence of hydrogen on the high-cycle fatigue resistance of austenitic stainless steels in a hydrogen environment within the temperature range of 150K (-120°C) to 373K (100°C).

The material in this study is a type 304L austenitic stainless steel grade. Due to differences in stacking fault energy compared to more stable stainless steel grades, such as 316L, this steel grade is expected to exhibit varying proportions of martensitic transformation at low temperatures under fixed loading conditions compared to other steel grades [4], which may influence susceptibility to HE [5].

The objective of this paper is to examine the effect of temperature on fatigue properties of tests conducted in air and to establish a correlation between the decrease in fatigue life of tests conducted in gaseous hydrogen at ambient temperature for lifetimes ranging from 10^4 to 2×10^6 cycles. The paper presents the results of a selected austenitic stainless steel grade, followed by the experimental conditions. The results section covers the ratcheting behaviour and fatigue strength, with the discussion exploring the interaction between the two and the impact of temperature and hydrogen.

2. MATERIALS AND EXPERIMENTALS PROCESS

TABLE 1 : CHEMICAL COMPOSITIONS OF 304L STAINLESS STEEL (wt%)

| | C | Si | Mn | P | S | Ni | Cr |
|------|-------|-------|------|-------|-------|------|------|
| 304L | 0.024 | 0.328 | 1.88 | 0.034 | 0.001 | 8.08 | 18.1 |

The material investigated in this study is a 304L type austenitic stainless steel. Its chemical composition is given in TABLE 1. The material was supplied in the form of a rolled plate 18 mm thick. Cylindrical fatigue specimens were taken in the rolling direction. The diameter in the gage length was 4.5 mm and the length was 15 mm. Cylindrical test specimens were machined for fatigue testing and polished to achieve a mirror-like surface finish, with an arithmetic mean roughness (R_a) of less than 0.02 μm .

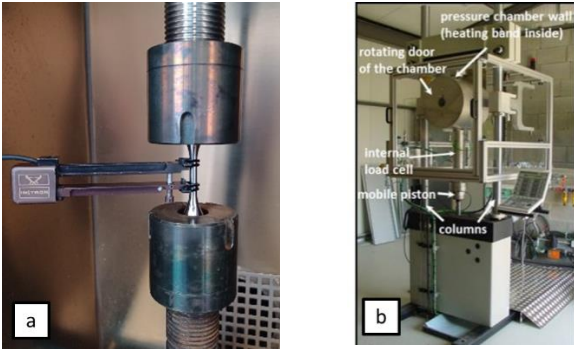


FIGURE 1 : (a) CYLINDRICAL SAMPLE FOR TEST AND (b) IN AIR HYCOMAT 1 HYDROGEN TEST RIG.

FIGURE 1a. presents the test conducted in air. Prior to testing, it was confirmed that there was no self-heating at the given test conditions. It is used to perform mechanical tests in a hydrogen gas environment at different temperatures. The hydrogen gas pressure inside the pressurized chamber was 1.5 MPa so as to be representative of the pressures inside liquid hydrogen storage tanks.

First, monotonic tensile tests were carried out on cylindrical specimens with a gauge length of 20 mm and a diameter of 4 mm using an MFX 500B extensometer in accordance with the NF EN ISO 9513 standard. These tensile tests were carried out at a constant strain rate of 10^{-3}s^{-1} , measured. These tests were carried out at different temperatures ($-83^{\circ}\text{C} < T < 100^{\circ}\text{C}$) in order to assess the reference properties of the material. To achieve low temperatures, the sample was tested in an autoclave cooled with liquid nitrogen. The specimen temperature was measured by a thermo-couple attached on the specimen surface at middle of gage length. The results are summarised in TABLE 2. The material undergoes a significant increase in strength when exposed to low temperatures.

TABLE 2 : TENSILE PROPERTIES AT DIFFERENT TEMPERATURES IN AIR.

| Temperature (°C) | Yield stress $R_{p0,2}$ (MPa) | Ultimate tensile strength R_m (MPa) | Elongation A(%) |
|------------------|-------------------------------|---------------------------------------|-----------------|
| -83 | 556 | 1330 | 45 |
| 25 | 336 | 700 | 95 |
| 100 | 313 | 726 | 45 |

The fatigue test conditions are show in TABLE 3. The tests were stress controlled with a load ramp for the first 200 cycles to avoid loading above the maximum load (overshoot). The criterion used to select the stress levels for the study was to ensure that the fatigue life remained between 10^4 and 2.10^6 . The evolution of strain was measured using an extensometer. To evaluate the ratcheting behaviour, the average strain ϵ_m and stress σ_m were considered. Fatigue tests were performed until total failure of specimens or run-out at two million cycles. Observations of fracture surfaces were conducted on fractured specimens to assess damage mechanisms and their sensitivity to the presence of hydrogen. The fracture surfaces were observed by using a scanning electron microscope (SEM).

TABLE 3 : NUMBER OF TEST CONDUCTED AND FATIGUE CONDITIONS

| Environment | Number of test conducted | Load ratio | Frequency (Hz) | Fatigue life range | Gas pressure (MPa) |
|----------------|--------------------------|------------|----------------|---------------------|--------------------|
| Air, 25°C | 21 | 0,1 | 10 | $10^4 > N > 2.10^6$ | 0,1 |
| Air, 100°C | 13 | | | | 0,1 |
| Air, -83°C | 8 | | | | 0,1 |
| Hydrogen, 25°C | 14 | | | | 1,5 |

3. RESULTS

1. RATCHETTING

The average deformation curves as a function of the number of cycles for 304L at different temperatures in air (a) and in a hydrogen environment at room temperature (b) are shown in FIGURE 2. The mean stress and strain values for a fatigue cycle are represented by σ_m and ϵ_m , respectively. These values are defined as the average between the maximum and minimum values for each cycle. It is important to note that the parameter with the greatest effect on the ratchetting deformation is temperature. Tests carried out at room temperature (25°C) and at higher temperatures (100°C) were subjected to the same average stress. To compare the results obtained at 25°C with those obtained at -83°C, we have chosen the lifetime (N_f) as the indicator for comparison. The increase in exposure temperature results in an increase in average strain during the first few cycles, with a lower progressive strain rate at lower temperatures. At low temperatures, for an equivalent life at ambient temperature, the final strain (ϵ_f) is lower than at higher temperatures, which is explained by the hardening of the material as a function of the lower temperature.

The results of tests carried out in a hydrogen environment at room temperature show no difference in ratchetting behaviour compared to tests carried out in air.

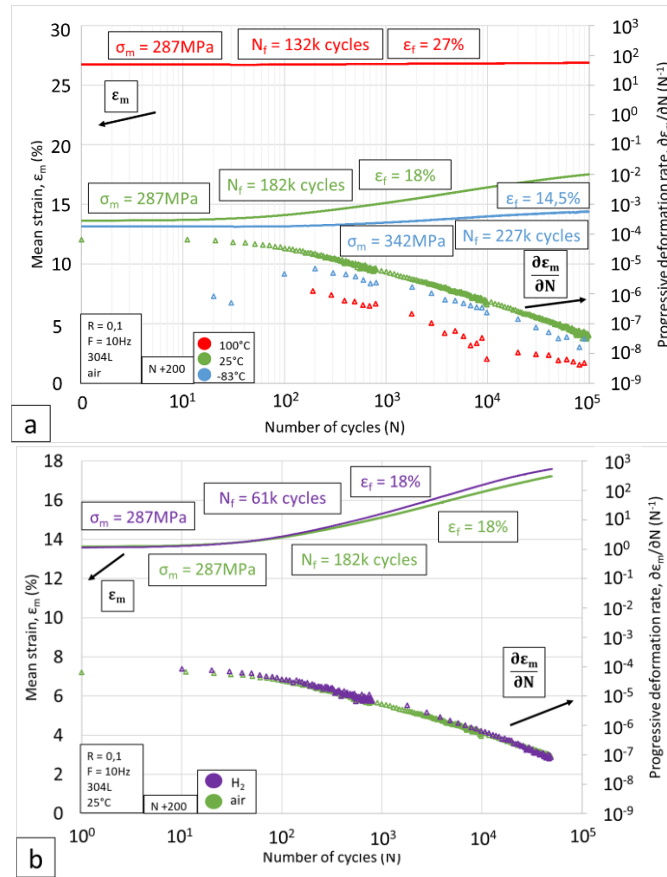


FIGURE 2 : EVOLUTION OF THE MEAN STRAIN AND AVERAGE DEFORMATION RATE AS A FUNCTION OF THE NUMBER OF CYCLES FOR 304L FOR 3 TEMPERATURES IN AIR (a) AND IN ENVIRONMENT (PH2 = 1,5 MPa) AT ROOM TEMPERATURE (b).

2. FATIGUE STRENGTH

FIGURE 3 shows the S-N curves for 304L at the three temperatures namely 100°C, 25°C, and -83°C. It appears that the lower the temperature, the higher the life is for a given stress amplitude. However, the difference in fatigue life is greater between T_{-83°C} and T_{25°C}. To estimated the fatigue life for high stress level, Basquin model [6] have been used:

$$\text{Log}(N) = C - m \cdot \log(\sigma_a) \quad (1)$$

With N the number of cycles, C, m two constants and σ_a is the stress amplitude. TABLE 4 show the value of the C and m coefficients.

TABLE 4 : BASQUIN PARAMETERS

| Test condition | C | m |
|----------------------|-------|-------|
| -83°C, air | 33,20 | 11,33 |
| 25°C, air | 44,6 | 16,55 |
| 100°C, air | 23,90 | 7,86 |
| 25°C, H ₂ | 92,50 | 36,86 |

The results of tests carried out in a hydrogen environment appear to indicate a reduction in fatigue life at ambient temperature for the highest stress levels. However, at lower stress levels the effect of hydrogen diminishes. Ogata [3] indicates an identical phenomenon on a SUS304L type steel at room temperature.

The slope of the Wöhler data differs between the fatigue life results of the air temperature tests. This is particularly noticeable for the S-N curve at 100°C. It would appear that at higher temperatures, the increase in average stress leads to a smaller reduction in fatigue life. However, because of the wide dispersion of lifetimes at the lowest stress levels, the endurance limit has not yet been clearly identified. Further tests at low stresses would be necessary to ascertain this limit.

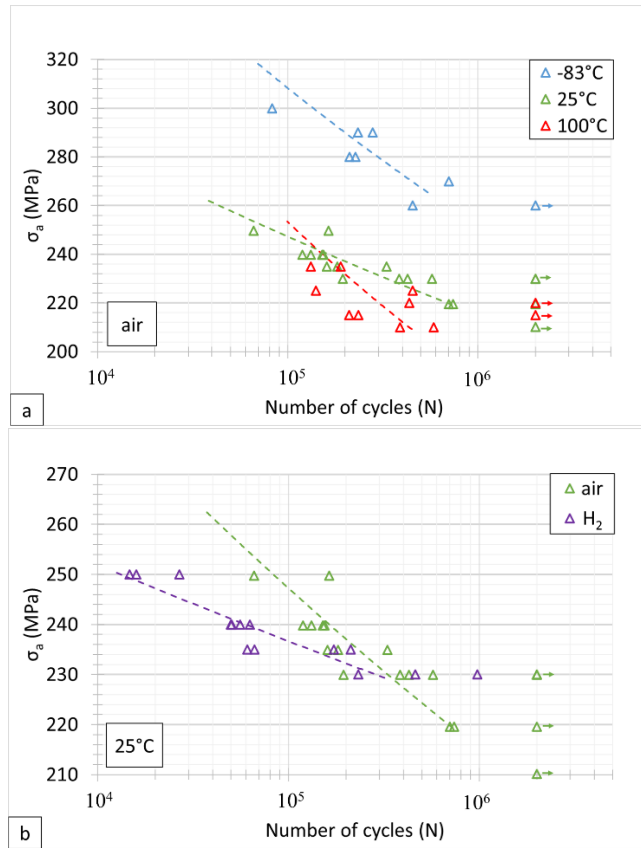


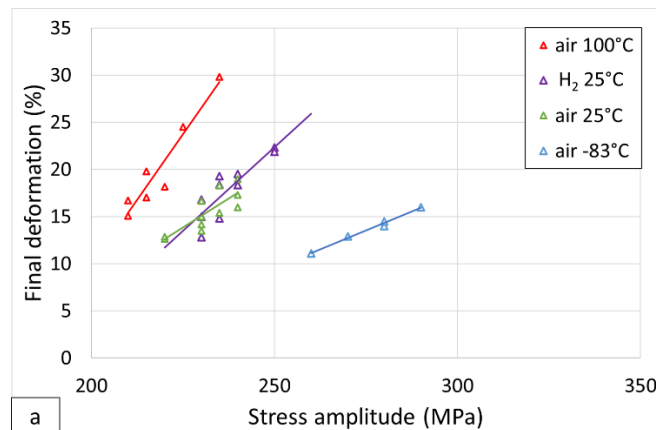
FIGURE 3 : S-N CURVE FOR 304L AT 3 DIFFERENT TEMPERATURES IN AIR (a) AND AT ROOM TEMPERATURE IN HYDROGEN (b).

4. DISCUSSION

Xia et al [7] demonstrated that the fatigue life of an ASME A516 low alloy steel depended on the mean positive stress and the ratcheting strain. The fatigue life decreased as the final ratcheting strain increased. However, by performing a preloading step before testing, the ratcheting deformation rate decreased by an order of magnitude for tests conducted at the highest stresses, resulting in an increase in fatigue life.

Figure 4 illustrates the impact of applied stress amplitude on the ultimate ratcheting deformation and the variation of the final deformation (the value correspond to the deformation reach at the end of the stable deformation) over the fatigue life. For each of the investigated temperatures, correlations are observed between stress amplitude and final ratcheting strain : the final ratcheting strain increases with the stress amplitude. At a given stress amplitude, an increase in temperature results in an increase of final ratcheting strain. With regard to the fatigue life, a clear correlation is observed between fatigue life and final ratcheting strain. The higher the final ratcheting strain ϵ_f , the lower the fatigue life N_f . Results obtained at 100°C show a different trend compared to other investigated temperature suggesting a potential change of mechanism.

The results of tests carried out in a hydrogen environment at room temperature show no difference in ratcheting deformation compared to tests carried out in air.



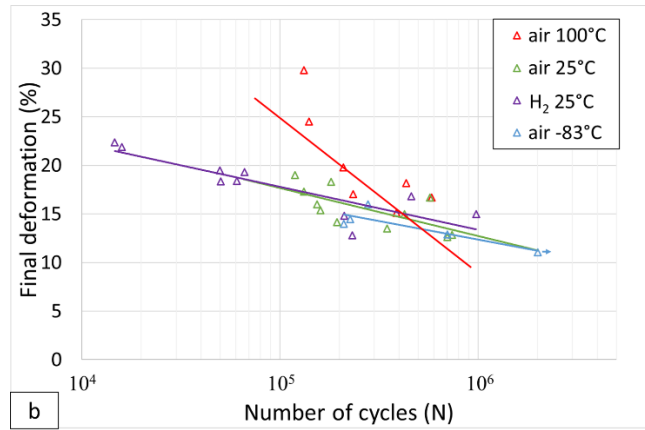


FIGURE 4 : EVOLUTION OF FINAL STRAIN AT FRACTURE, BASED ON STRESS AMPLITUDE (a) AND ON THE NUMBER OF CYCLES (b), FOR 304L IN BOTH AIR AND HYDROGEN ENVIRONMENT AT DIFFERENT TEMPERATURES

Fracture surface analysis can provide hints to assess the impact of hydrogen on crack initiation (primary and secondary), the propagation mode (intergranular, transgranular or mixed), and fatigue crack growth rate. Here SEM observations revealed an equal amount of initiation on inclusion-type defects in both environments. However, further observation and analysis are still necessary to obtain more representative results.

The effect of hydrogen on fatigue crack growth rate is well documented in literature. Significant effects were observed on 304 type stainless steel, see [9] for instance. In absence of conventional fatigue crack propagation test, fatigue crack growth rate is evaluated through fracture surface analysis. This analysis requires the use of a fracture surface form factor as introduced by Carpinteri [8]. Thus, the stress intensity factor K is calculated as follow :

$$K_{I,F} = k_{I,F} \cdot \sigma_F (\pi a)^{1/2} \quad (2)$$

with $k_{I,F}$ the form factor is approximately 0.95 for specimens broken at 100°C and approximately 1.5 for tests broken at -83°C., σ_F the nominal stress, and a the distance from initiation site.

FIGURE 5 illustrates the evolution of the fatigue crack growth rate da/dN as a function of the stress intensity factor ΔK . The crack propagation rate is slightly affected by temperature, but under hydrogen conditions, fatigue crack propagation rates are higher by a factor of 2 for similar ΔK values. These results are consistent with those reported by Matsunaga et al. during fatigue crack propagation in a grade of 304 steel [9], where an increase in fatigue crack growth rate of a factor of 5 was observed between a hydrogen and air environment, for tests performed at 1 Hz under a gas pressure of 0.7 MPa.

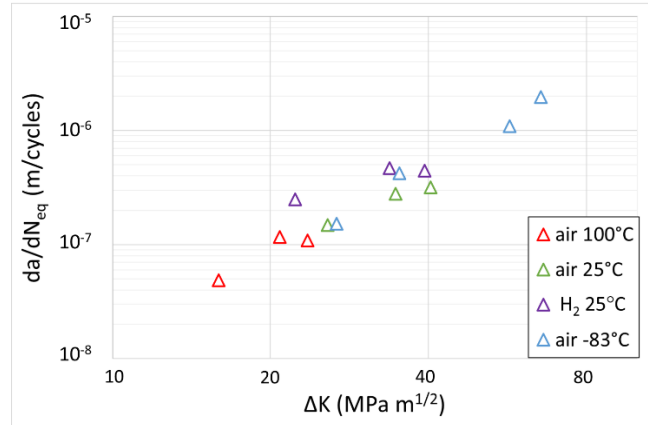


FIGURE 5 : RELATIONSHIP BETWEEN CRACK GROWTH RATE, DA/DN AND STRESS INTENSITY FACTOR RANGE, ΔK . THE VALUE OF ΔK CAN BE OBTAINED BY CONSIDERING MULTIPLE ZONES AT VARYING DISTANCES FROM THE INITIATION POINT.

FIGURE 6 displays two fracture surfaces resulting from fatigue tests conducted at -83°C and 100°C in air. Analysis of these surfaces indicates a significant morphological difference between the tests carried out at -83°C and those carried out at 100°C. The stable crack propagation depth at low temperatures is greater than that at higher temperatures. However, there was no difference in

the propagation mode between the surfaces produced at different temperatures. In the current state of analysis, no significant morphological difference between fracture surfaces of specimen tested in air and in hydrogen is observed.

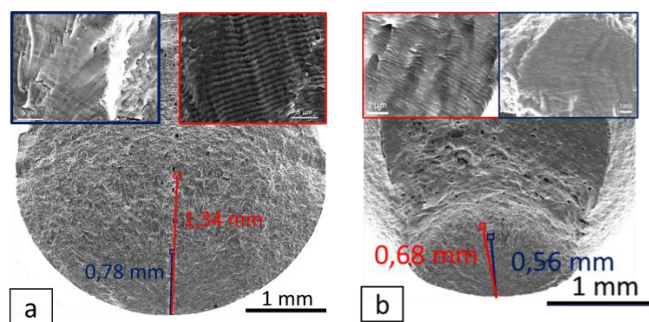


FIGURE 6 : FRACTURE SURFACES OF 304L TESTED AT -83°C (A) AND 100°C (B) IN AIR.

5. CONCLUSION

Based on the experimental results presented in this paper, it can be concluded that temperature significantly affects the fatigue properties of 304L austenitic stainless steel in air. Additionally, propagation distances in air are higher at lower temperatures.

At room temperature, no difference in ratcheting behaviour was observed in a hydrogen environment compared to air. However, in hydrogen environment, the fatigue life at the highest stress levels is reduced compared to tests carried out in air. Furthermore, hydrogen has been found to increase crack propagation speeds. Tests are planned to determine the impact of hydrogen on low-temperature fatigue life at -120°C.

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